



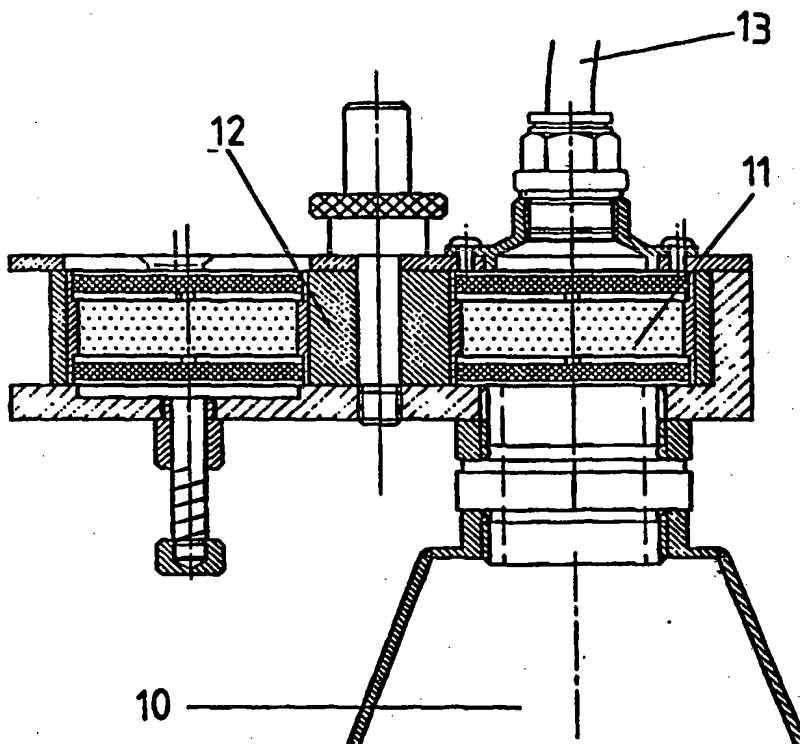
## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification n 6 : <b>G01N 1/40, 1/24</b>		A1	(11) International Publication Number: <b>WO 98/57141</b>
			(43) International Publication Date: 17 December 1998 (17.12.98)
(21) International Application Number: PCT/EP98/03531			(74) Agents: NILSSON, Brita et al.; AB Stockholm Patentbyrå Zacco & Bruhn, Sveavägen 170, P.O. Box 23101, S-104 35 Stockholm (SE).
(22) International Filing Date: 10 June 1998 (10.06.98)			<b>AH</b>
(30) Priority Data: 60/049,449 12 June 1997 (12.06.97) US 9702407-9 24 June 1997 (24.06.97) SE			
(71) Applicant (for all designated States except US): BIOSENSOR APPLICATIONS SWEDEN AB [SE/SE]; Skäpplandsgatan 1, S-703 46 Örebro (SE).			
(72) Inventors; and			
(75) Inventors/Applicants (for US only): GÄRDHAGEN, Peter [SE/SE]; Ringshyttans Gärd, Striberg, S-713 93 Nora (SE). SAMUELSSON, Magnus [SE/SE]; Närjeholmsvägen 26, S-633 46 Eskilstuna (SE). ALM, Arne [SE/SE]; Junivägen 4C, S-632 22 Eskilstuna (SE). JANSSON, Åke [SE/SE]; Rökstavägen 9, S-633 52 Eskilstuna (SE). ARNELL, Johan [SE/SE]; Bryggaregatan 10, S-633 43 Eskilstuna (SE). ANDERSSON, Claes [SE/SE]; Sandviksvägen 7A, S-691 52 Karlskoga (SE). JOHANSSON, Erik [SE/SE]; Stinavägen 66, S-691 54 Karlskoga (SE).			
(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, GM, GW, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).			
Published With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.			

(54) Title: APPARATUS, SYSTEM AND METHOD FOR THE DETECTION OF AN ANALYTE IN AIR

## (57) Abstract

An apparatus, a system and a method for the detection of an analyte in air comprising an air sampling system (1) comprising a mouthpiece (10) and a fan, a first enrichment stage (11) for enrichment of the analyte comprising a thermally regulated adsorption/desorption filter (20) creating a non-laminar gas flow for adsorption of the analyte, a heat source for heating the filter to evaporate the analyte, a second enrichment stage (30) for the further enrichment of the gaseous analyte comprising a cold trap (32) for condensing the analyte, extraction of the condensed material with a solvent, and collecting the solution, and analyzing the collected liquid sample with an analytical detection system, are disclosed. Further, an adsorption/desorption filter creating a non-laminar gas flow comprising a winding (21) of a resistance wire coated with an active material electric heating of the wire, is described. A specific application is detection of a gaseous component of explosives or mines, e.g. TNT, in air.



**FOR THE PURPOSES OF INFORMATION ONLY**

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AL	Albania	ES	Spain	LS	Lesotho	SI	Slovenia
AM	Armenia	FI	Finland	LT	Lithuania	SK	Slovakia
AT	Austria	FR	France	LU	Luxembourg	SN	Senegal
AU	Australia	GA	Gabon	LV	Latvia	SZ	Swaziland
AZ	Azerbaijan	GB	United Kingdom	MC	Monaco	TD	Chad
BA	Bosnia and Herzegovina	GE	Georgia	MD	Republic of Moldova	TG	Togo
BB	Barbados	GH	Ghana	MG	Madagascar	TJ	Tajikistan
BE	Belgium	GN	Guinea	MK	The former Yugoslav Republic of Macedonia	TM	Turkmenistan
BF	Burkina Faso	GR	Greece	ML	Mali	TR	Turkey
BG	Bulgaria	HU	Hungary	MN	Mongolia	TT	Trinidad and Tobago
BJ	Benin	IE	Ireland	MR	Mauritania	UA	Ukraine
BR	Brazil	IL	Israel	MW	Malawi	UG	Uganda
BY	Belarus	IS	Iceland	MX	Mexico	US	United States of America
CA	Canada	IT	Italy	NE	Niger	UZ	Uzbekistan
CF	Central African Republic	JP	Japan	NL	Netherlands	VN	Viet Nam
CG	Congo	KE	Kenya	NO	Norway	YU	Yugoslavia
CH	Switzerland	KG	Kyrgyzstan	NZ	New Zealand	ZW	Zimbabwe
CI	Côte d'Ivoire	KP	Democratic People's Republic of Korea	PL	Poland		
CM	Cameroon	KR	Republic of Korea	PT	Portugal		
CN	China	KZ	Kazakhstan	RO	Romania		
CU	Cuba	LC	Saint Lucia	RU	Russian Federation		
CZ	Czech Republic	LI	Liechtenstein	SD	Sudan		
DE	Germany	LK	Sri Lanka	SE	Sweden		
DK	Denmark	LR	Liberia	SG	Singapore		
EE	Estonia						

## **APPARATUS, SYSTEM AND METHOD FOR THE DETECTION OF AN ANALYTE IN AIR**

5

The present invention relates to an apparatus, a system and a method for the detection of an analyte in air. The invention is particularly concerned with the detection of dangerous chemicals such as explosives, mines, bombs, war gases and narcotics.

10

### **Background**

15

It is difficult to selectively detect small amounts of a gaseous analyte in air, and it is particularly important to do so when the location of dangerous chemicals is to be determined.

20

For example, since World War II, mine detection has been mostly carried out with the help of metal seekers that detect small amounts of metal contained in most mines. As a result of the development of anti-personal mines with less and less metal, the sensitivity of a metal seeker has been enhanced to give alarm for less than one gram of metal. However, at this sensitivity level metal fragments in the ground will block its use in most mined terrain by giving a false alarm.

25

Another method for mine detection, and narcotics detection, which has been successfully applied, has been the use of trained dogs. But even the best dog can only work for short periods with full attention and with long rest periods in between. Well trained dogs are also very expensive.

30

It would be desirable to have an apparatus, a system and a method for the selective detection of small amounts of an analyte in air for many applications,

**CONFIRMATION COPY**

e. g. in the process industry for the detection of specific contamination or leakage, in the airport safety control, customs and police work for the detection of narcotics and bombs, and in clearing the terrain of mines and bombs after a war situation.

5

### Description of the invention

The present invention provides an apparatus, a system and a method for the selective detection of an analyte in a sample of air. The sample of air is  
10 collected at a suspected location. The possible specific analyte is enriched from the air sample in accordance with the present invention, and finally the specific analyte of interest is analyzed from a small amount of liquid.

One aspect of the invention is concerned with an apparatus for the detection of  
15 an analyte in air comprising  
an air sampling system (1) for collecting an air sample comprising a mouthpiece (10) and a fan,  
means for transport of the air through a first enrichment stage (11) for enrichment of the analyte comprising a thermally regulated  
20 adsorption/desorption filter creating a non-laminar gas flow for adsorption of the analyte on the filter surfaces, encased in a housing having a gas inlet and a gas outlet connected to valves, and a heat source for heating the filter to a temperature high enough to evaporate the analyte but low enough to avoid decomposition of the analyte,  
25 means for transport of the desorbed analyte from the filter to a second enrichment stage for the further enrichment of the gaseous analyte comprising a cold trap (32) for condensing the analyte, means for extraction of the condensed material with a solvent, and means for collecting the solution enriched in analyte from the cold trap, and  
30 means for transport of the collected liquid sample to an analytical detection system for the detection of the analyt .

In a preferred embodiment of this aspect of the invention, the valves are substituted for a revolver, the first enrichment stage is reconditioned by cooling gas, and the second enrichment stage is connected to a revolver and  
5 reconditioned by washing the cold trap with a solvent.

Another aspect of the invention is concerned with an adsorption/desorption filter creating a non-laminar gas flow, which comprises a gas inlet and a gas outlet connected to a housing (20) in which a winding (21) of a resistance wire,  
10 such as a copper wire, coated with an active (insulating) material having affinity for an analyte to be tested, such as Teflon®, is encased, and two connector bands (22) attached to the ends of the wire for electric heating of the wire to a temperature that provides a temperature on the surface of the active material at which the analyte is desorbed without decomposition.

15 A further aspect of the invention is concerned with a system for the detection of an analyte in air comprising  
an air sampling system comprising a mouthpiece and a fan,  
a first enrichment stage for enrichment of the analyte comprising a thermally  
20 regulated adsorption/desorption filter creating a non-laminar gas flow comprising a housing having a gas inlet and a gas outlet connected to valves, and  
a heat source for heating the filter to a temperature high enough to evaporate the analyte but low enough to avoid decomposition of the analyte,  
25 a second enrichment stage for the further enrichment of the analyte comprising a cold trap for condensing the analyte, extraction of the condensed material with a solvent, and collecting the solution enriched in analyte from the cold trap, and  
an analytical detection system for the detection of the analyte from a liquid  
30 sample.

In a preferred embodiment of this aspect of the invention, the valves are substituted for a revolver, the first enrichment stage is reconditioned by cooling gas, and the second enrichment stage is connected to a revolver and reconditioned by washing the cold trap with a solvent.

5

Yet another aspect of the invention is concerned with a method of detecting an analyte in air comprising  
sampling of air with an air sampling system comprising a mouthpiece and a fan,  
passing the air to a first enrichment stage where the analyte is adsorbed on the  
10 surfaces of a thermally regulated adsorption/desorption filter creating a non-laminar gas flow,  
heating the filter surfaces to a temperature high enough to evaporate the analyte but low enough to avoid decomposition of the analyte,  
flushing the filter with a small amount of an inert gas, such as nitrogen or air,  
15 passing the vapor from the first enrichment stage to a second enrichment stage for the further enrichment of the analyte by condensation in a cold trap,  
flushing the cold trap with a small amount of solvent to dissolve the condensed material,  
collecting the small amount of solution containing the analyte in an container,  
20 and  
analyzing the presence of analyte in the container with the aid of an analytical detection system for the detection of the analyte in liquid sample.

25

In a preferred embodiment the analyte is a gaseous component of explosives or mines, such as TNT (2,4,6- trinitrotoluene), RDX (1,3,5-trinitro-1,3,5-triazacyclohexane) or PETN (pentaerythritoltetranitrate).

30

The invention is also concerned with the use of the apparatus and system, respectively, each according to the invention, for the detection of a gaseous component of explosives or mines, e.g. TNT (2,4,6- trinitrotoluene), RDX (1,3,5-trinitro-1,3,5-triazacyclohexane) or PETN (pentaerythritoltetranitrate), and use

of the adsorption/desorption filter according to the invention in an apparatus, system and method for the detection of an analyte in air.

The invention will now be illustrated with the aid of the following description of specific embodiments, related drawings and examples. However, the invention should not be considered to be limited to such exemplification.

An apparatus, a system and a method of the invention preferably comprises the following elements

• 1) An Air Sampling System comprising

- mouth piece
- dust filter (optional)
- hygrometer (optional)
- fan ( or external pump)
- injection valve (preferred embodiment)

• 2) A First Enrichment Stage comprising

- adsorption/desorption filter of active (insulating) (e.g. hydrophobic) material
- valves (revolver)
- heater
- filter reconditioning

• 3) A Second Enrichment Stage comprising

- cold trap
- solution
- pump (optionally manual injection)
- washing (reconditioning of cold trap)

6

- 4) One or more analytical detection system(s), such as a) gas chromatograph or b) biosensor(s) (each) comprising

- flow cell
- quartz crystal
- surface antibody coating, the antibody being specific for and binding to
- the specific analyte of interest

- 5) Optional Signal Processing comprising, in the case of 4) b),

- reference oscillator
- CPU
- serial communication link

- 6) Optional Man Machine Interface comprising

- acoustic alarm
- presentation LEDs
- control switch

- 7) Optional Support Systems for each of the elements 1 to 6, comprising

- structure
- power supply

The method of the invention starts with the sampling of air, which possibly contains or is suspected to contain a specific analyte of interest, with air sampling.

#### **Air Sampling System.**

Air is collected with the help of a fan (or ejector pump), through a mouthpiece held at a suspected location, such as just over the ground of a suspected mine, and the air is transported, if necessary, through a filter for dust separation, to the first enrichment stage and through its adsorption/desorption filter to an



exhaust facing away from the ground. The air sampling system may also include a calibration and test system, optionally controlled from the signal processing, for automated injection of a small, calibrated amount of analyte, e.g. TNT ( 2,4,6- trinitrotoluene) vapor or TNT solution, into the airflow ahead of the first enrichment stage. The air sampling system should have a neutral surface, or surface coating, with regard to the analyte content of the air stream, at least for the duration of one sample in the sampling system (gilding has been used in prototype).

- 10 Preferably, the moisture content of the air inflow has to be known, and sometimes adjusted, to facilitate the subsequent process control. A hygrometer may be placed within the air inlet or in connection with the air sampling system, for instance on the outside of the mouthpiece.
- 15 **The First Enrichment Stage** operates with an (e.g. hydrophobic) adsorption/-desorption filter which should be of a material (etched polytetrafluoroethylene, Teflon®, used in prototype) that, with high efficiency, adsorbs the analyte , such as TNT, from air at the normal sampling temperature, and desorbs it again when it is heated to a higher temperature, which however should be lower than the decomposition temperature of the analyte (e. g. TNT).
- 20

In an embodiment, the construction of the filter, in the first enrichment stage, is made from an etched hydrophobic and electrically insulating material deposited on metal wire. This has many advantages: The etched material gives a very high effective area for adsorption and the metal wire core allows very fast electrical heating for desorption. In addition, the use of a hydrophobic material will allow most unwanted chemicals and water-vapor to pass through the first stage, and only hydrophobic vapors are trapped by the filter.

- 25
- 30 During the adsorption stage the air flow through the filter is driven by the fan (pump) in the air sampling system. During the desorption stage the transport

and vaporization of the analyte, such as TNT, in the filter is driven by a small amount of an inert gas together with the heat expansion of the trapped air, thus giving an enrichment of analyte (e.g. TNT) vapor in the process gas (dry nitrogen was used in prototype).

5

The adsorption-desorption filter in the first enrichment stage will function for a limited number of temperature cycles and must therefore be replaced at regular service intervals.

10

**The Second Enrichment Stage** contains a (e.g. hydrophilic) cold trap (quartz at ambient temperature) for condensation of the analyte (e.g. TNT) vapor from the first enrichment stage, and a pump system for the transport of a drop of a solvent, such as water, through the cold trap into a container for analysis. The type of the container depends on the type of method and equipment selected for the analysis. In case piezoelectric determination is selected, the container may be one or several biosensor cell(s).

15

20

In case the analyte is TNT, the water solution will normally not accept such a hydrophobic chemical, but TNT is polar enough to have an acceptable solubility at the actual low concentrations. The use of a hydrophilic second enrichment stage thus eliminates most of the disturbing hydrophobic chemicals that passed the first enrichment stage.

#### **Analytical detection system**

25

The condensed material in the cold trap is flushed with a small amount of solvent to dissolve the material, and the solution containing the analyte is collected in a container, e.g. an air sampling tube of a gas chromatograph or a flow cell of a biosensor system, depending on which type of analytical detection system is to be used.

30

In the prototype, the cold trap is manually washed with buffer solution, which is then injected into the container for analysis on a gas chromatograph or a biosensor system.

- 5 Gas chromatographs are standard laboratory equipment, and a biosensor system which has been used in Example 2 in the Experimental part of this description is described below.

### **Biosensor system**

- 10 The Biosensor system used in the piezoelectric determination of the analyte, comprises a flow cell, in which the solvent/water solution, from the second enrichment stage, passes over the surface of an oscillating quartz crystal, coated with a layered structure exposing an antibody complex active against the analyte, e.g. TNT, to the solution. The antibodies bind to the analyte, e.g.
- 15 TNT, in the solution, and the weight difference, at the surface of the crystal, gives a slight change in the resonance frequency in the electronic circuit incorporating the crystal. When the number of antibodies available for binding to the analyte is reduced due to the complex formation with the analyte in the test solution, the cell ceases to function. Then, the quartz crystal is replaced
- 20 with another one which has a fresh coating. In an automated embodiment, this situation will be detected by a signal processing unit, and an automatic or manual change-over to a fresh flow cell takes place.

- The drain pipe from one biosensor flow cell may be directly connected to the
- 25 inlet of another, thus adding sensitivity to a different analyte, such as an explosive or a narcotic, or a multisensor system of parallel biosensors can be arranged by dividing the inlet into an array of flow cells.

- The crystal in the flow cell may be operated in such a way, that a pump function
- 30 of the solution will accrue, thus eliminating the need for a separate pump in the second enrichment stage.

The Signal Processing Electronics contain electronics for operating the crystal, measurement of different parameters, control of the whole enrichment procedure and measurement of process and decision algorithms for the evaluation of the results. More precisely, it measures continuously the changes in the resonance frequency of the crystal(-s) in the biosensor(-s) and makes the necessary calculations to evaluate if the analyte, e.g. TNT, vapor is present in the tested air sample, and presents the result to the operator or operative system. To measure the small frequency changes in the biosensor, a high short to medium time precision time/frequency reference, in form of an OCXO (Oven Controlled Crystal Oscillator) or better, must be used. (Standard precision lab instrument was used in prototype).

The Central Processing Unit in the signal processing electronics will also supervise the power supply, the function of the biosensor, make automatic tests and calibrations, at start-up and at biosensor shifts, and evaluate and signal when the useful life of a biosensors is consumed.

The signal processing electronics will include a serial communication link, for communication with an external computer in a multi-sensor system, for detection and localization of analyte, or for testing, control and service.

The Man Machine Interface (MMI) is the operator's normal way of communication with the signal processing electronics when the biosensor system is used as a stand alone equipment, and no control commands are executed through the serial communication link. Presentation of equipment status and the results of the analysis (one type of sensors in use) are presented by 3 signal lamps or LED:s of different color and an acoustic warning signal from a beeper or through an earphone. A red lamp may be used to indicate detection of the analyte vapor and a green for a clear, i.e. that nothing is detected by the biosensor. In between there may also be a yellow lamp

indicating an indifferent status, e.g. that more information/sampling is necessary for a reliable indication either way. Acoustic alarm shall accompany a rise in the risk – color shift from green to yellow (red) or yellow to red.

- 5 The operator's settings on the MMI are on-off, continuous-intermittent measurement and a measurement-0-test trigger.

In the prototype a PC (personal computer) was used for signal processing and the MMI simulated on its screen.

10

The support systems are the mechanical and the electrical structure of the unit and the power supply with batteries and voltage converters/regulators.

15

The mechanical structure may be a light, two-part design for one man, a backpack and a hand-held sniffer part. The backpack, with batteries and most of the electronics of the system, is connected via a cable to the sniffer part, which is hand-held but with a supporting body harness. Thus, the complete unit is a small light-weight unit that can be worn during a whole working day with low stress from the wearing.

20

The power pack can take batteries of different style, both primary and rechargeable. Power can also be fed from an outside (12 V DC) source. The recommended power source for field use, as a stand alone equipment, is primary cells of the "D"-size.

25

The support system for the demonstrator is standard laboratory and PC equipment.

30

The above disclosed biosensor system is designed to be the main sensor(-s) in single- or multisensor systems for the detection of an analyte in an air sample collected at a suspected location. The biosensor system is highly sensitive and

selective and can be designed to detect vapor from a single analyte when antibodies directed to said analyte are used as in the coating of the biosensor cell. The biosensor system may also contain several biosensors for detection of different analytes.

5

The disclosed biosensor system is designed to be as sensitive as a dog for the smell from the analyte but with higher reliability and availability at a reasonable cost.

## 10 Description of the drawings

**Fig. 1** shows a cross-section of an air sampling system for collecting an air sample. A mouthpiece (10) is held at the suspected location, e.g. just above the ground, and the air is transported through an adsorption filter (11) of a first enrichment stage with the aid of an air pump, which is connected (13) to the backside of the filter, thus being less sensitive to contamination. After each sampling, the filter is moved to its next position (desorption) by a revolver (12);

15

**Fig. 2 a.** shows a cross-section of a first enrichment stage comprising a adsorption-desorption filter. The filter comprises a winding (21) of a resistance wire, such as a metal wire, coated with an active (insulating) material having affinity for the tested analyte (e.g. a hydrophobic material) encased in a plastic housing (20) and with two circular connector bands (22) soldered to the ends of the wire,

20

**Fig. 2 b.** shows a perspective view of the first enrichment stage, where the solder joint can be seen;

25

**Fig 3 a.** shows a side view of the parts of a second enrichment stage comprising a cold trap ( e.g. of a hydrophilic material). The cold trap is composed of a quartz capillary (32) in a heat sink of stone (31) mounted in a plastic housing (30). The whole unit is held together by a single screw (34)

30

cooperating with an elastic (rubber) distance pin. An O-ring (33) is also mounted at the tip of the capillary (32) as a seal to the adsorption filter from the first enrichment stage,

Fig 3 b. shows a cross-section of the second enrichment stage;

Fig 4. shows a schematic presentation of the function of a filter heater. To desorb the analyte (TNT) from the filter, the temperature is increased from ambient temperature to the desorption temperature. The heating is effected in a very short time, and the temperature is then held constant until the desorption is finished. This can be done without the use of a separate temperature sensor (which might disturb the adsorption of the analyte, TNT) by using the resistance (e.g. copper) wire in the filter as a thermometer. For example, copper has a good and well known temperature constant, i.e. the resistance in the filter wire will increase with the temperature. If the filter is operated with a known constant current, it is possible to measure the voltage drop in the winding and get a voltage potential to the temperature.

In fig 4 a., the following functions are shown,

- (1) the copper in the filter wire is heated to the desorption temperature,
- (2) a constant current is generated,
- (3) measurement of raw signal,
- (4) the measurement of the raw signal is converted to a voltage which is proportional to the temperature in the filter, and it is adapted to control of the pulse wide modulator (PWM),
- (5) a trigger signal indicates to the signal processing when the filter temperature shall be measured (heating current off),
- (6) PWM signal for high power control (with the switch in off position only when the constant measurement current goes through the wire), and
- (7) main heating current to filter wire.

Fig. 4 b., shows schematically each of the signals (1) - (7).

Fig. 5a + 5b., shows a schematic presentation of an automated system for air sampling and enrichment of analyte according to the invention.

## EXPERIMENTS

- 5 A prototype of the above disclosed automated sampling system was use for the detection of TNT as the analyte.

### Example 1

- 10 A test amount of TNT was placed in a plastic bag which was fastened with tape on the inside wall of a suitcase.

A mouthpiece (10) was stuck into the suitcase, and air was sucked from the suitcase and passed through the first enrichment stage, i.e. an  
15 absorption/desorption filter (20).

#### Adsorption:

- |               |   |
|---------------|---|
| Suction time: | 10 sec.   |
| Flow rate :   | 45 L /min.  |
| Source :      | Suitcase, 50 L, with 10 g of TNT in a plastic bag |
| 20 Filter :   | Copper wire with a plasma etched Teflon® coating. |

- Thermal desorption, i.e electric current through the wire of the filter, liberates the TNT adsorbed on the surface of the wire coating, and during the desorption nitrogen gas is passed through the filter and carries the TNT vapors to the  
25 second enrichment stage, i.e. a cold trap, where TNT is condensed on the walls of the capillary (32).

#### Desorption:

- |                                     |           |
|-------------------------------------|-----------|
| Desorption temperature:             | 200 °C    |
| Desorption flow (N <sub>2</sub> ) : | 20 ml/min |
| 30 Temperature in the cold trap:    | 25 °C     |
| Material in the cold trap:          | quartz    |



The cold trap was emptied with the aid of a buffer solution which was introduced by injection with a syringe. The buffer solution dissolves the TNT. Then the solution was sucked from the cold trap with an injection syringe.

5

Buffer solution : 0.01 m Na-phosphate buffer, 0.1 M NaCl, pH= 7.4

Buffer volume: 10  $\mu$ l

Time for emptying : 1 minute

10 Analysis of TNT content by gas chromatography

Analysis of the TNT content in the cold trap was performed with a gas chromatograph having electron capture detection (GC-ECD).

15

The solution from the cold trap was placed on a wad of quartz wool in an air sampling tube. Before the injection started, the solvent was evaporated from the tube. Injection of samples were made by thermal desorption of the air sampling tube, simultaneously trapping the desorbed material in a cold trap, which subsequently was flash heated (thermal desorption cold trap injector, TCT).

20

**Instrument parameters:**

Instrument: Chrompack CP-9001 GC integrated with CP 4001  
TCT/PTI

GC column: 10 m x 0.32 mm CP-Sil 5 CB (1.2  $\mu$ m)

Carrier gas: Helium, 45 kPa

25

**TCT conditions:**

Cold trap: -70 °C

Desorption temp.: 250 °C

Desorption time: 15 min

Desorption flow: 35 ml/min

30

Injection temp.: 200 °C

Injection time: 1 min

GC conditions:

1 min at 150 °C, followed by 150 °C → 250 °C, 10 °C/min

5      Detector : Electron capture detector (ECD)

Results: the yield of TNT was approximately 200 pg (relative standard deviation 30%)

10      **Example 2**

The Example 1 was repeated but the analysis of the TNT content in the cold trap was performed with a biosensor system as described in the general part of this description.

15

A piezoelectric biosensor having a coating on the crystal comprising antibodies' binding specifically to TNT was used for the detection of TNT in the solution from the cold trap.

Results: TNT could still be detected at the low level of 10 picograms.

20

**Example 3**

The sampling system described in Example 1 was used for determination of the influence of the desorption time on the total efficacy.

25      The test parameters were as follows

Adsorption:

Suction time:            30 sec.

Flow rate     :            approx. 1L /sec.

Source        :            Wooden box with 10 g of TNT

30      Filter        :            Copper wire with a plasma etched Teflon® coating.

**Desorption:**

Desorption temperature: 200 °C

Desorption flow (N<sub>2</sub>) : 20 ml/min

Heating time: 3 sec.

5 Desorption time: 3, 18 or 120 sec.

**Emptying of the cold trap:**

Solvent: Acetone

Volume added: 3 µl

Volume extracted: approx. 1.5 µl (the rest had evaporated)

10

**Analysis:**

Analysis on GC- ECD (cf. Example 1) (Split injection)

**Results:**

15 The results are summarized in Table 1.

**Table 1.**

Analyzed contents of TNT at different desorption times

Desorption time	TNT content (ng)
3	0.45
3	0.61
18	0.87
18	1.04
120	0.82
120	0.99

20

The results in Table 1 indicate that a desorption time of 18 sec. is sufficient for obtaining the same yield as at longer desorption times. Already at 3 sec. 60% of the yield is obtained. An optimization of the parameters should result in a reduction of the desorption time to at most 18 sec. with retained efficacy.

**Example 4**

This experiment was conducted for the purpose of finding out if the air above  
5 real mines buried in the ground detecting contains detectable amounts of TNT.

The samples were collected in South Africa in the vicinity of an anti-tank mine  
(AT mine) with the aid of an absorption/desorption filter according to the  
invention inserted into an air sampling equipment of Mechchem.

10 Samples were taken at a position just above the mine, walking past the mine  
and along a section of road which had been classified as mine free. (The AT  
mine was also placed on a section of the road). The absorption/desorption  
filters were sent to us in Sweden for analysis. The sampling was made in a  
15 blind way, i.e. without giving the knowledge to us before analysis.

Adsorption:

Suction time: 1, 2 or 4 min.

Flow rate : approx. 1L /sec.

Source : see above

20 Filter : Copper wire with a plasma etched Teflon® coating.

Desorption:

Desorption temperature: 200 °C

Desorption flow (N<sub>2</sub>) : 20 ml/min

25 Desorption time: 60 sec.

Emptying of the cold trap:

Solvent: Acetone

Volume added: 3 µl

30 Volume extracted: approx. 1.5 µl (the rest had evaporated)

**Analysis:**

Analysis from acetone on GC- ECD (cf. Example 1) (Split injection)

**Results:**

- 5 The results are summarized in Table 2.

**Table 2.**

Summary of the results from the tests in South Africa

Sample	TNT content (pg)	Collected volume of air (liter)	TNT content per collected volume of air (pg/L)
AT mine, just above	150	60	2.5
AT mine, walking by	330	120	2.7
Mine free road	30	240	0.1

- 10 The reason for the low content of TNT on the mine free section of road is not known, but may be due to contamination during transport of the filters to Sweden.

- 15 These initial results from real mine testing show that it is possible to decide which section of a road is mine free with the detection system using air sampling and an adsorption/desorption filter of the invention.

### Claims

- 5       **1.** Apparatus for the detection of an analyte in air comprising  
an air sampling system (1) for collecting an air sample comprising a  
mouthpiece (10) and a fan,  
means for transport of the air through a first enrichment stage (11) for  
enrichment of the analyte comprising a thermally regulated adsorption/-  
10       desorption filter creating a non-laminar gas flow for adsorption of the analyte  
on the filter surfaces, encased in a housing having a gas inlet and a gas outlet  
connected to valves, and a heat source for heating the filter to a temperature  
high enough to evaporate the analyte but low enough to avoid decomposition  
of the analyte,  
15       means for transport of the desorbed analyte from the filter to a second  
enrichment stage for the further enrichment of the gaseous analyte comprising  
a cold trap (32) for condensing the analyte, means for extraction of the  
condensed material with a solvent, and means for collecting the solution  
enriched in analyte from the cold trap, and  
20       means for transport of the collected liquid sample to an analytical detection  
system for the detection of the analyte.
- 25       **2.** Apparatus for the detection of an analyte in air according to claim 1, wherein  
the valves are substituted for a revolver, the first enrichment stage is  
reconditioned by cooling gas, and the second enrichment stage is connected to  
a revolver and reconditioned by washing the cold trap with a solvent.
- 30       **3.** Adsorption/desorption filter creating a non-laminar gas flow, which comprises  
a gas inlet and a gas outlet connected to a housing (20) in which a winding (21)  
of a resistance wire coated with an active (insulating) material having affinity  
for an analyte to be tested is encased, and two connector bands (22) attached

to the ends of the wire for electric heating of the wire to a temperature that provides a temperature on the surface of the active material at which the analyte is desorbed without decomposition.

5

4. Adsorption/desorption filter according to claim 3, wherein the resistance wire is a copper wire and the coating material is Teflon®.

10

5. System for the detection of an analyte in air comprising an air sampling system comprising a mouthpiece and a fan, a first enrichment stage for enrichment of the analyte comprising a thermally regulated adsorption/desorption filter creating a non-laminar gas flow comprising a housing having a gas inlet and a gas outlet connected to valves, and

15 a heat source for heating the filter to a temperature high enough to evaporate the analyte but low enough to avoid decomposition of the analyte, a second enrichment stage for the further enrichment of the analyte comprising a cold trap for condensing the analyte, extraction of the condensed material with a solvent, and collecting the solution enriched in analyte from the cold trap,

20 and an analytical detection system for the detection of the analyte from a liquid sample.

25

6. System for the detection of an analyte in air according to claim 1, wherein the valves are substituted for a revolver, the first enrichment stage is reconditioned by cooling gas, and the second enrichment stage is connected to a revolver and reconditioned by washing the cold trap with a solvent.

30

7. Method of detecting an analyte in air comprising sampling of air with an air sampling system comprising a mouthpiece and a fan, passing the air to a first enrichment stage where the analyte is adsorbed on the

surfaces of a thermally regulated adsorption/desorption filter creating a non-laminar gas flow,

heating the filter surfaces to a temperature high enough to evaporate the analyte but low enough to avoid decomposition of the analyte,

5 flushing the filter with a small amount of an inert gas,  
passing the vapor from the first enrichment stage to a second enrichment stage for the further enrichment of the analyte by condensation in a cold trap,  
flushing the cold trap with a small amount of solvent to dissolve the condensed material,

10 collecting the small amount of solution containing the analyte in an container,  
and  
analyzing the presence of analyte in the container with the aid of an analytical detection system for the detection of the analyte in liquid sample.

15 8. Method of detecting an analyte in air according to claim 7, wherein the analyte is a gaseous component of explosives or mines.

9. Use of the apparatus according to any one of the claims 1-3, for the detection of a gaseous component of explosives or mines.

20

10. Use of the adsorption/desorption filter according to claim 4, in an apparatus, system and method for the detection of an analyte air.

11. Use of the system according to any one of the claims 5 and 6, for the  
25 detection of a gaseous component of explosives or mines.



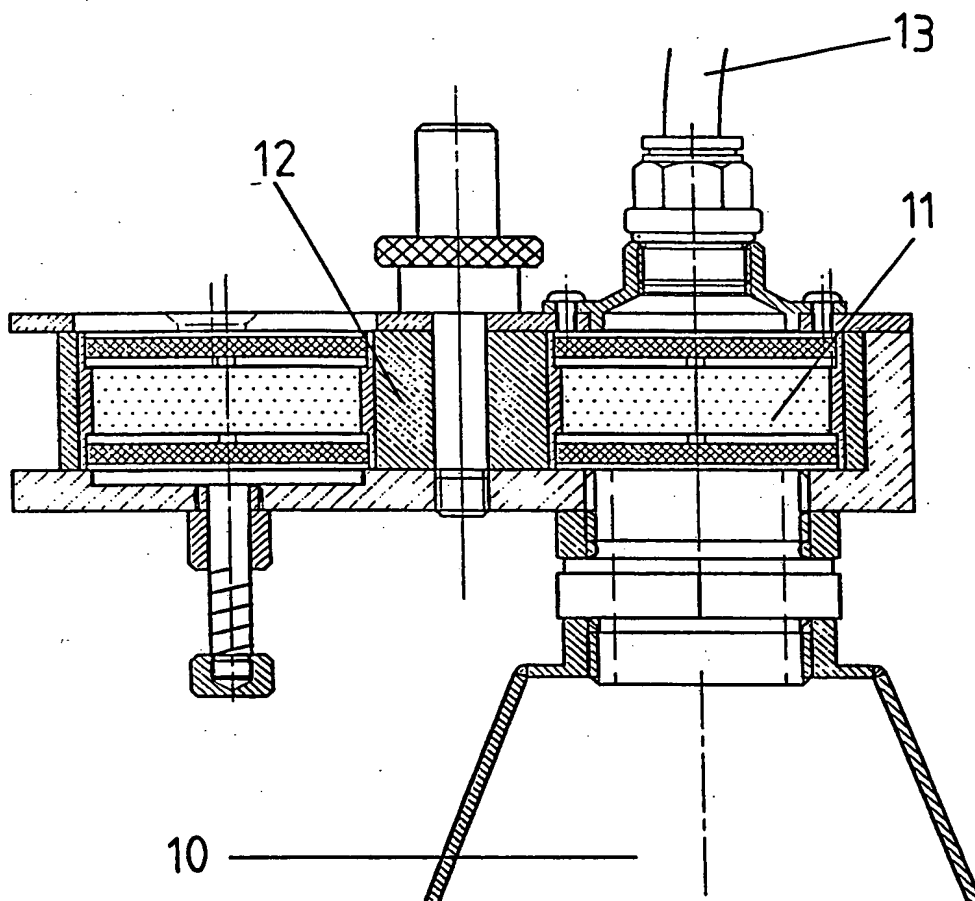


Fig. 1

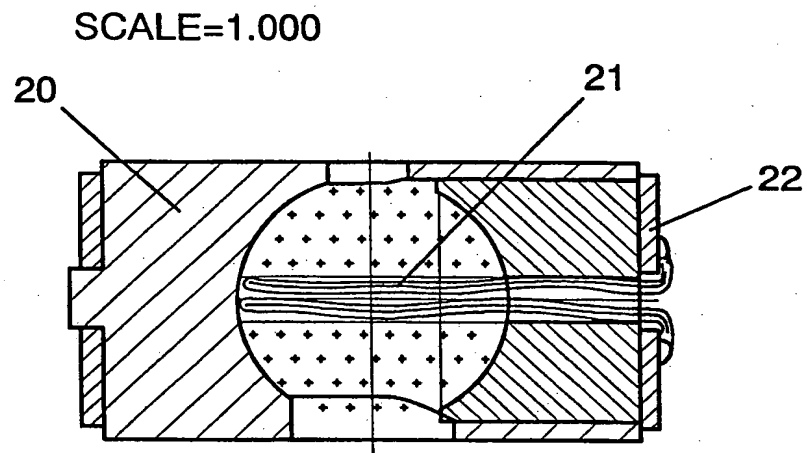


Fig. 2a

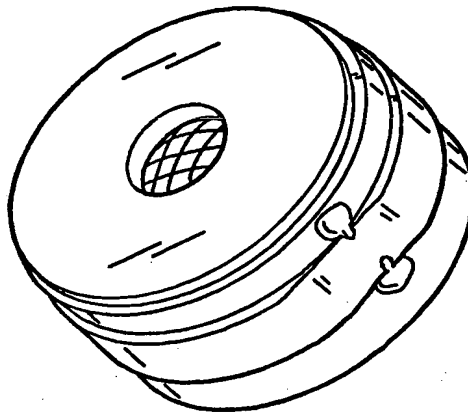
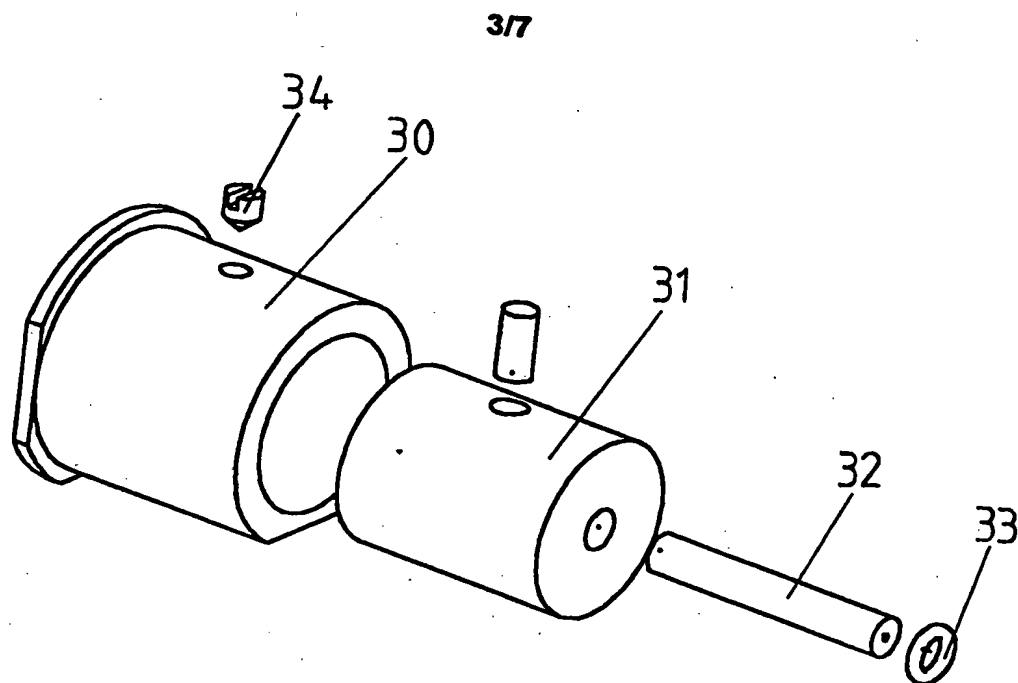
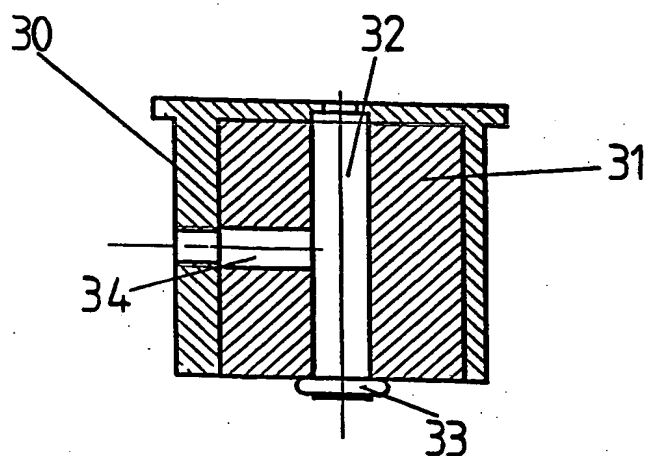


Fig. 2b

SUBSTITUTE SHEET (RULE 26)

**Fig. 3a****Fig. 3b**

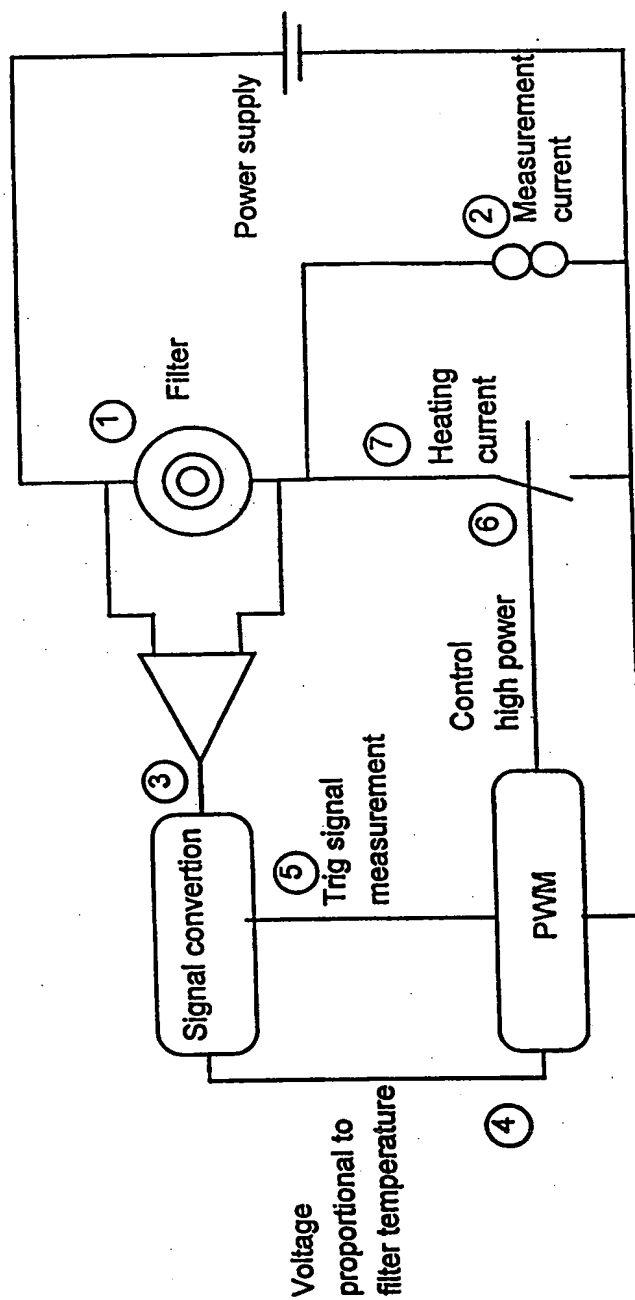


Fig. 4a

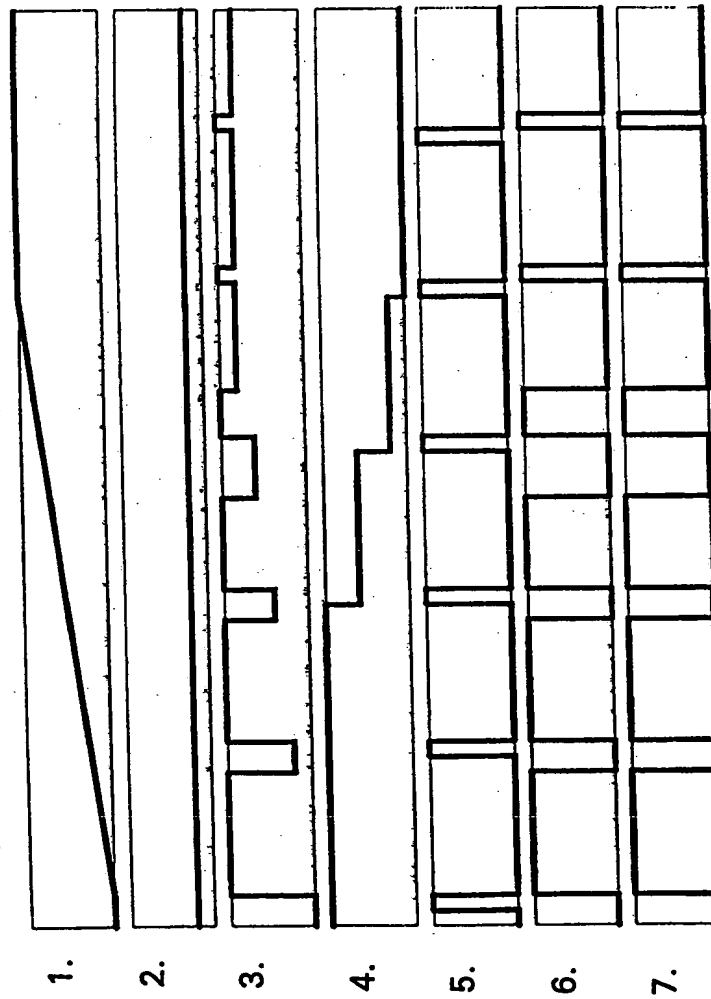


Fig. 4b

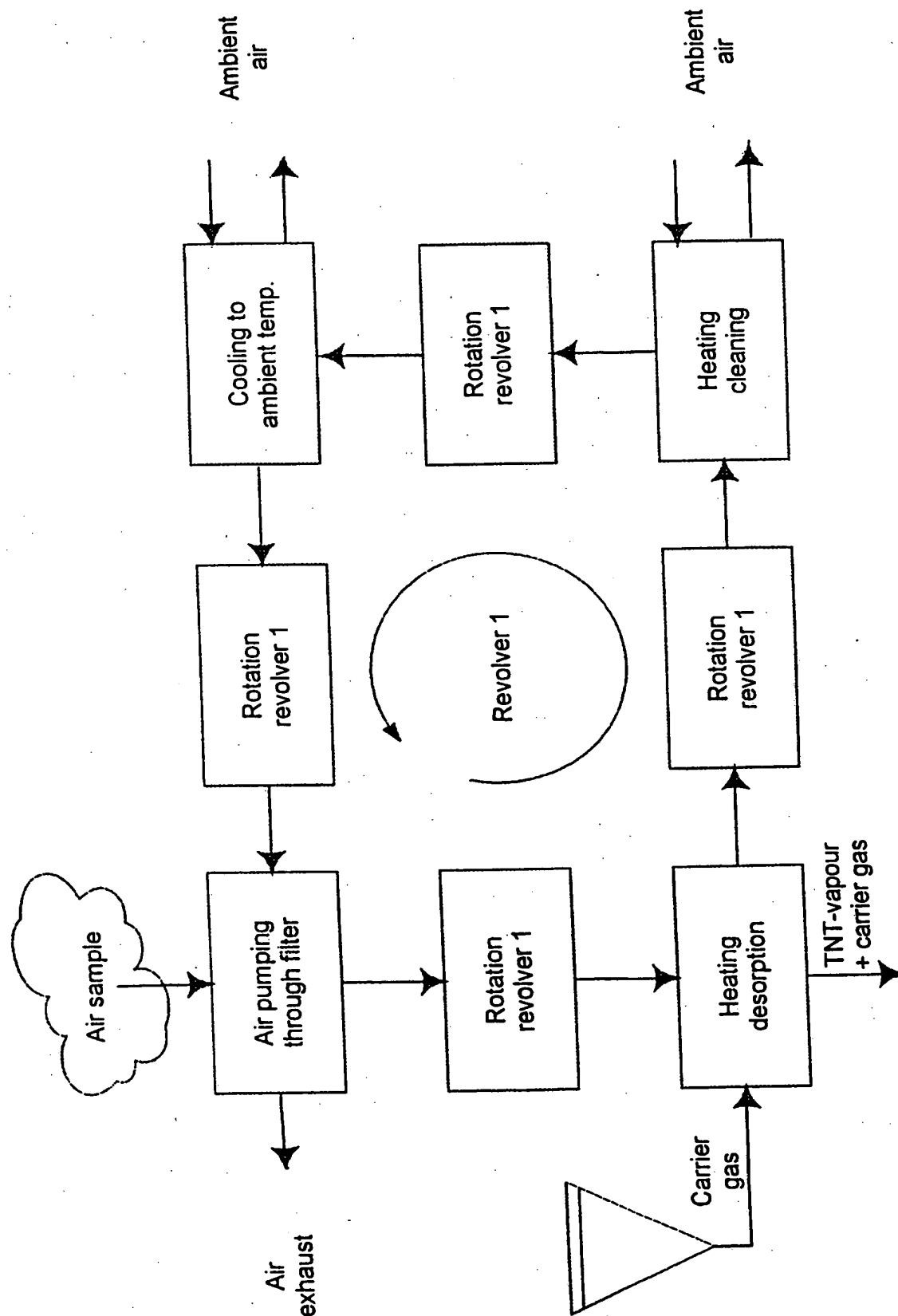


Fig. 5a

7/7

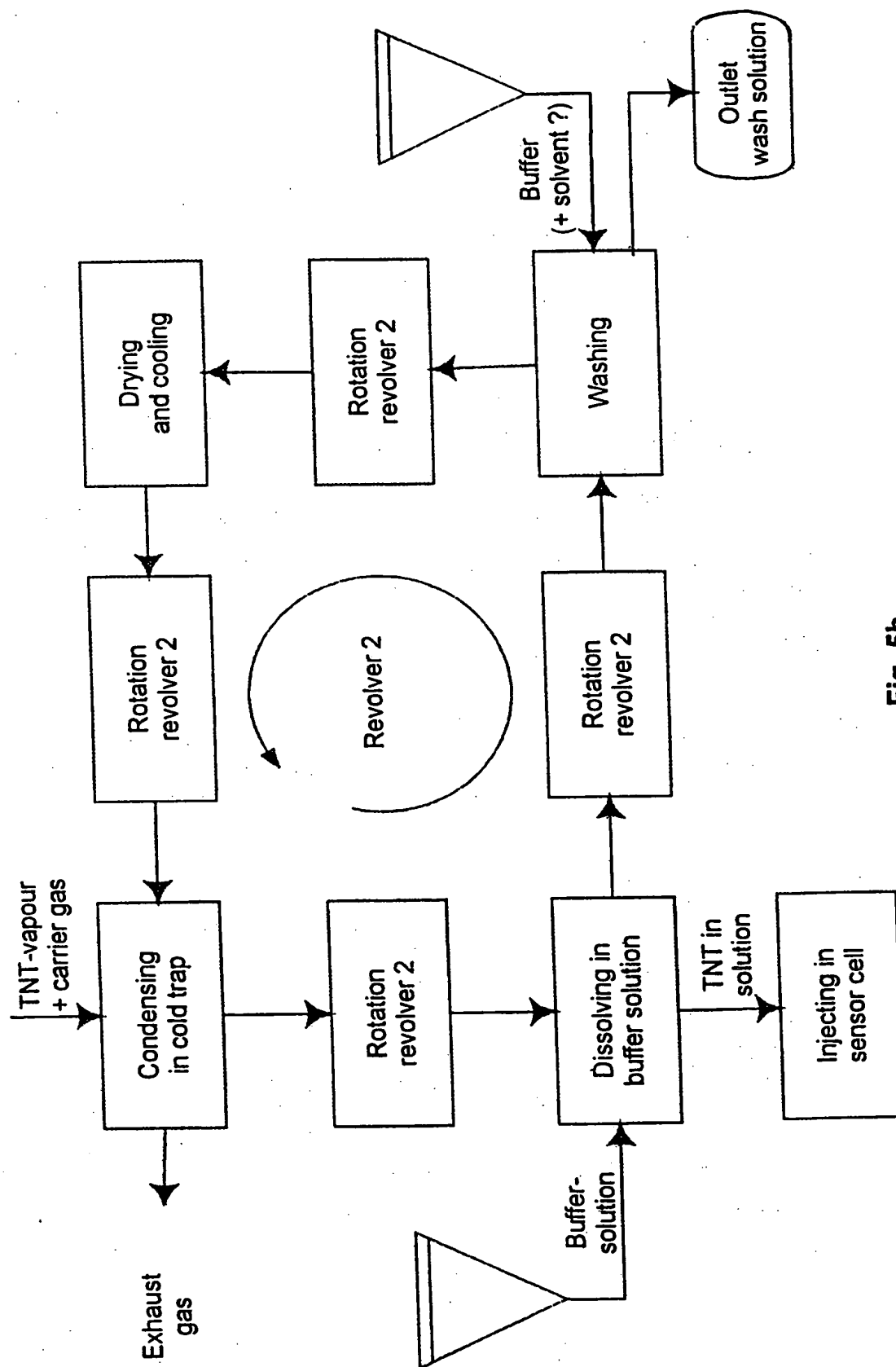


Fig. 5b

# INTERNATIONAL SEARCH REPORT

International Application No.

CT/EP 98/03531

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 G01N1/40 G01N1/24

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 G01N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 5 551 278 A (ROUNBEHLER DAVID P ET AL) 3 September 1996 see column 4, line 28 - line 39; figure 1 see column 6, line 50 - column 7, line 13; figure 8 see column 7, line 48 - line 55 see column 9, line 50 - line 60 see column 10, line 53 - column 11, line 50; figure 14	2
Y	see column 12, line 25 - line 44; figure 16	1, 5, 7-9, 11
Y	see column 13, line 10 - line 50; figure 16	1, 5
	--- -/--	

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

### \* Special categories of cited documents :

- "A" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier document but published on or after the international filing date
- "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- "O" document referring to an oral disclosure, use, exhibition or other means
- "P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search

6 November 1998

Date of mailing of the international search report

27/11/1998

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2  
NL - 2280 HV Rijswijk  
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,  
Fax: (+31-70) 340-3016

Authorized officer

Hocquet, A



# INTERNATIONAL SEARCH REPORT

International Application No

PCT/EP 98/03531

## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	EP 0 401 861 A (RES CORP TECHNOLOGIES INC) 12 December 1990 see page 7, line 54 - page 9, line 5; figure 7 see page 9, line 41 - page 10, line 29; figures 7,10	2,6
Y	US 4 194 884 A (FINE DAVID H ET AL) 25 March 1980 see column 1, line 11 - line 48 see column 3, line 55 - line 60 see column 5, line 57 - line 63 see column 6, line 25 - line 30 see column 8, line 6 - line 34 see column 10, line 1 - line 8	1,2,5-9, 11
A	US 4 819 477 A (FISHER WILLIAM H ET AL) 11 April 1989	2,6
A	see column 7, line 29 - column 8, line 39	3
A	see column 9, line 19 - line 42	3
A	PATENT ABSTRACTS OF JAPAN vol. 016, no. 575 (C-1011), 15 December 1992 & JP 04 227014 A (MATSUI MFG CO), 17 August 1992 see abstract	1,2,5,6
A	US 5 300 758 A (ROUNBEHLER DAVID P ET AL) 5 April 1994 see figure 1	1,5
A	US 4 128 008 A (LINENBERG AMOS) 5 December 1978 see figures	2,6
A	US 5 138 889 A (CONRAD FRANK J) 18 August 1992 see figures	1,5
X	US 5 395 589 A (NACSON SABATINO) 7 March 1995 see column 6, line 37 - column 7, line 10; figures 2,4	3
A	FR 2 660 873 A (GRASEBY IONICS LTD) 18 October 1991 see page 7, line 35 - page 8, line 4	4
A	US 4 909 090 A (MCGOWN JAMES B ET AL) 20 March 1990	1,2
A	see column 1, line 31 - line 42	1,2
X	see column 3, line 43 - line 52	3
	-/--	

# INTERNATIONAL SEARCH REPORT

Int .tional Application No "

PCT/EP 98/03531

## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	<p>JOSEFSON ET AL.: "adsorption of organic compounds . . ."</p> <p>ANALYTICAL CHEMISTRY,</p> <p>vol. 56, 1984, pages 764-768, XP002083559</p> <p>us</p> <p>see the whole document</p> <p>-----</p>	4

# INTERNATIONAL SEARCH REPORT

on patent family members

ional Application No

EP 98/03531

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 5551278 A	03-09-1996	US 5092217 A	03-03-1992
		US 5099743 A	31-03-1992
		WO 9108466 A	13-06-1991
		US 5092219 A	03-03-1992
		US 5092155 A	03-03-1992
		US 5092218 A	03-03-1992
		US 5092157 A	03-03-1992
		US 5092156 A	03-03-1992
		US 5123274 A	23-06-1992
		US 5310681 A	10-05-1994
		US 5098451 A	24-03-1992
		EP 0501955 A	09-09-1992
EP 0401861 A	12-12-1990	US 4987767 A	29-01-1991
		AT 153131 T	15-05-1997
		AU 633582 B	04-02-1993
		AU 5690590 A	13-12-1990
		CA 1329494 A	17-05-1994
		CA 2018697 A	09-12-1990
		DE 69030686 D	19-06-1997
		DE 69030686 T	11-09-1997
		DK 401861 T	03-11-1997
		ES 2102355 T	01-08-1997
		GR 3024358 T	28-11-1997
		IL 94684 A	29-12-1994
		IL 107672 A	15-03-1995
		JP 2119373 C	06-12-1996
		JP 3087629 A	12-04-1991
		JP 8033338 B	29-03-1996
		JP 2610118 B	14-05-1997
		JP 7209151 A	11-08-1995
		KR 9409049 B	29-09-1994
		US 5465607 A	14-11-1995
		US 5585575 A	17-12-1996
		US 5345809 A	13-09-1994
		AU 679387 B	26-06-1997
		AU 3063495 A	09-11-1995
		AU 7032291 A	18-07-1991
		CA 2070648 A	09-06-1991
		EP 0502998 A	16-09-1992

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No.

PCT/EP 98/03531

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP 0401861 A		JP 9126964 A JP 9126965 A JP 9126966 A WO 9109307 A US 5109691 A	16-05-1997 16-05-1997 16-05-1997 27-06-1991 05-05-1992
US 4194884 A	25-03-1980	DE 2944774 A FR 2442073 A GB 2035125 A,B JP 55074440 A NL 7907940 A SE 7909685 A	12-06-1980 20-06-1980 18-06-1980 05-06-1980 28-05-1980 25-05-1980
US 4819477 A	11-04-1989	CA 1279207 A EP 0236047 A JP 6100521 B JP 63079033 A	22-01-1991 09-09-1987 12-12-1994 09-04-1988
US 5300758 A	05-04-1994	US 5108705 A AT 134445 T CA 2037862 A DE 69117201 D DE 69117201 T EP 0447174 A JP 4220558 A	28-04-1992 15-03-1996 13-09-1991 28-03-1996 19-09-1996 18-09-1991 11-08-1992
US 4128008 A	05-12-1978	NONE	
US 5138889 A	18-08-1992	NONE	
US 5395589 A	07-03-1995	CA 2120682 A GB 2288234 A,B	07-10-1995 11-10-1995
FR 2660873 A	18-10-1991	GB 2243447 A,B	30-10-1991
US 4909090 A	20-03-1990	CA 2015157 A EP 0395194 A JP 2296128 A	24-10-1990 31-10-1990 06-12-1990



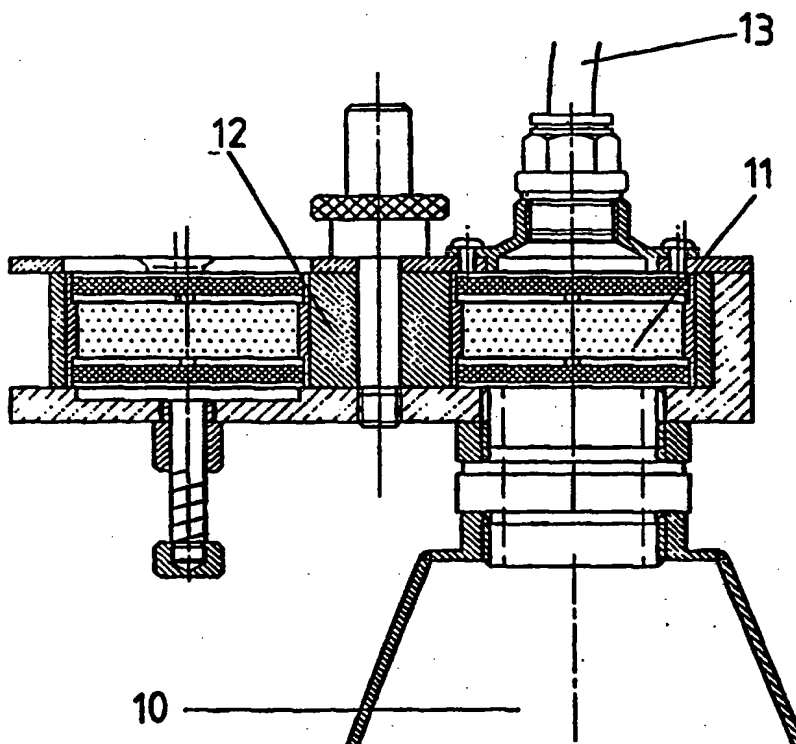
INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification <sup>6</sup> : <b>G01N 1/40, 1/24</b>		A1	(11) International Publication Number: <b>WO 98/57141</b>
			(43) International Publication Date: 17 December 1998 (17.12.98)
(21) International Application Number: PCT/EP98/03531		(74) Agents: NILSSON, Brita et al.; AB Stockholm Patentbyrå Zacco & Bruhn, Sveavägen 170, P.O. Box 23101, S-104 35 Stockholm (SE).	
(22) International Filing Date: 10 June 1998 (10.06.98)			
(30) Priority Data: 60/049,449 12 June 1997 (12.06.97) US 9702407-9 24 June 1997 (24.06.97) SE		(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, GM, GW, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).	
(71) Applicant (for all designated States except US): BIOSENSOR APPLICATIONS SWEDEN AB [SE/SE]; Skäpplandsgatan 1, S-703 46 Örebro (SE).			
(72) Inventors; and (75) Inventors/Applicants (for US only): GÅRDHAGEN, Peter [SE/SE]; Ringshyttans Gård, Striberg, S-713 93 Nora (SE). SAMUELSSON, Magnus [SE/SE]; Närjeholmsvägen 26, S-633 46 Eskilstuna (SE). ALM, Arne [SE/SE]; Junivägen 4C, S-632 22 Eskilstuna (SE). JANSSON, Åke [SE/SE]; Rökstavägen 9, S-633 52 Eskilstuna (SE). ARNELL, Johan [SE/SE]; Bryggaregatan 10, S-633 43 Eskilstuna (SE). ANDERSSON, Claes [SE/SE]; Sandviksvägen 7A, S-691 52 Karlskoga (SE). JOHANSSON, Erik [SE/SE]; Stinavägen 66, S-691 54 Karlskoga (SE).		Published With a revised version of the international search report. (88) Date of publication of the revised version of the international search report: 4 February 1999 (04.02.99)	

(54) Title: APPARATUS, SYSTEM AND METHOD FOR THE DETECTION OF AN ANALYTE IN AIR

(57) Abstract

An apparatus, a system and a method for the detection of an analyte in air comprising an air sampling system (1) comprising a mouth-piece (10) and a fan, a first enrichment stage (11) for enrichment of the analyte comprising a thermally regulated adsorption/desorption filter (20) creating a non-laminar gas flow for adsorption of the analyte, a heat source for heating the filter to evaporate the analyte, a second enrichment stage (30) for the further enrichment of the gaseous analyte comprising a cold trap (32) for condensing the analyte, extraction of the condensed material with a solvent, and collecting the solution, and analyzing the collected liquid sample with an analytical detection system, are disclosed. Further, an adsorption/desorption filter creating a non-laminar gas flow comprising a winding (21) of a resistance wire coated with an active material electric heating of the wire, is described. A specific application is detection of a gaseous component f x-plosives or mines, e.g. TNT, in air.



**FOR THE PURPOSES OF INFORMATION ONLY**

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AL	Albania	ES	Spain	LS	Lesotho	SI	Slovenia
AM	Armenia	FI	Finland	LT	Lithuania	SK	Slovakia
AT	Austria	FR	France	LU	Luxembourg	SN	Senegal
AU	Australia	GA	Gabon	LV	Latvia	SZ	Swaziland
AZ	Azerbaijan	GB	United Kingdom	MC	Monaco	TD	Chad
BA	Bosnia and Herzegovina	GE	Georgia	MD	Republic of Moldova	TG	Togo
BB	Barbados	GH	Ghana	MG	Madagascar	TJ	Tajikistan
BE	Belgium	GN	Guinea	MK	The former Yugoslav Republic of Macedonia	TM	Turkmenistan
BF	Burkina Faso	GR	Greece	ML	Mali	TR	Turkey
BG	Bulgaria	HU	Hungary	MN	Mongolia	TT	Trinidad and Tobago
BJ	Benin	IE	Ireland	MR	Mauritania	UA	Ukraine
BR	Brazil	IL	Israel	MW	Malawi	UG	Uganda
BY	Belarus	IS	Iceland	MX	Mexico	US	United States of America
CA	Canada	IT	Italy	NE	Niger	UZ	Uzbekistan
CF	Central African Republic	JP	Japan	NL	Netherlands	VN	Viet Nam
CG	Congo	KE	Kenya	NO	Norway	YU	Yugoslavia
CH	Switzerland	KG	Kyrgyzstan	NZ	New Zealand	ZW	Zimbabwe
CI	Côte d'Ivoire	KP	Democratic People's Republic of Korea	PL	Poland		
CM	Cameroon	KR	Republic of Korea	PT	Portugal		
CN	China	KZ	Kazakhstan	RO	Romania		
CU	Cuba	LC	Saint Lucia	RU	Russian Federation		
CZ	Czech Republic	LI	Liechtenstein	SD	Sudan		
DE	Germany	LK	Sri Lanka	SE	Sweden		
DK	Denmark	LR	Liberia	SG	Singapore		
EE	Estonia						

# INTERNATIONAL SEARCH REPORT

International Application No

PCT/EP 98/03531

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 G01N1/40 G01N1/24

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 G01N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 5 551 278 A (ROUNBEHLER DAVID P ET AL) 3 September 1996 see column 4, line 28 - line 39; figure 1 see column 6, line 50 - column 7, line 13; figure 8 see column 7, line 48 - line 55 see column 9, line 50 - line 60 see column 10, line 53 - column 11, line 50; figure 14	2
Y	see column 12, line 25 - line 44; figure 16	1,5,7-9, 11
Y	see column 13, line 10 - line 50; figure 16	1,5
	---	
	-/--	



Further documents are listed in the continuation of box C.



Patent family members are listed in annex.

### \* Special categories of cited documents:

- "A" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier document but published on or after the international filing date
- "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- "O" document referring to an oral disclosure, use, exhibition or other means
- "P" document published prior to the international filing date but later than the priority date claimed

- "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- "&" document member of the same patent family

Date of the actual completion of the international search

6 November 1998

Date of mailing of the international search report

27/11/1998

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2  
NL - 2280 HV Rijswijk  
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,  
Fax: (+31-70) 340-3016

Authorized officer

Hocquet, A

## INTERNATIONAL SEARCH REPORT

Int. .tional Application No

PCT/EP 98/03531

## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	EP 0 401 861 A (RES CORP TECHNOLOGIES INC) 12 December 1990 see page 7, line 54 - page 9, line 5; figure 7 see page 9, line 41 - page 10, line 29; figures 7,10 ---	2,6
Y	US 4 194 884 A (FINE DAVID H ET AL) 25 March 1980 see column 1, line 11 - line 48 see column 3, line 55 - line 60 see column 5, line 57 - line 63 see column 6, line 25 - line 30 see column 8, line 6 - line 34 see column 10, line 1 - line 8 ---	1,2,5-9, 11
A	US 4 819 477 A (FISHER WILLIAM H ET AL) 11 April 1989 ---	2,6
A	see column 7, line 29 - column 8, line 39	3
A	see column 9, line 19 - line 42 ---	3
A	PATENT ABSTRACTS OF JAPAN vol. 016, no. 575 (C-1011), 15 December 1992 & JP 04 227014 A (MATSUI MFG CO), 17 August 1992 see abstract ---	1,2,5,6
A	US 5 300 758 A (ROUNBEHLER DAVID P ET AL) 5 April 1994 see figure 1 ---	1,5
A	US 4 128 008 A (LINENBERG AMOS) 5 December 1978 see figures ---	2,6
A	US 5 138 889 A (CONRAD FRANK J) 18 August 1992 see figures ---	1,5
X	US 5 395 589 A (NACSON SABATINO) 7 March 1995 see column 6, line 37 - column 7, line 10; figures 2,4 ---	3
A	FR 2 660 873 A (GRASEBY IONICS LTD) 18 October 1991 see page 7, line 35 - page 8, line 4 ---	4
A	US 4 909 090 A (MCGOWN JAMES B ET AL) 20 March 1990 see column 1, line 31 - line 42 see column 3, line 43 - line 52 ---	1,2 1,2 3
	---	

-/--



# INTERNATIONAL SEARCH REPORT

Int .tional Application No

PCT/EP 98/03531

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	<p>JOSEFSON ET AL.: "adsorption of organic compounds . . ."</p> <p>ANALYTICAL CHEMISTRY,</p> <p>vol. 56, 1984, pages 764-768, XP002083559</p> <p>us</p> <p>see the whole document</p> <p>-----</p>	4

# INTERNATIONAL SEARCH REPORT

International application No.  
PCT/EP 98/03531

## Box I Observations where certain claims were found unsearchable (Continuation of item 1 of first sheet)

This International Search Report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:  
because they relate to subject matter not required to be searched by this Authority, namely:
  
2. ☐ Claims Nos.:  
because they relate to parts of the International Application that do not comply with the prescribed requirements to such an extent that no meaningful International Search can be carried out, specifically:
  
3. ☐ Claims Nos.:  
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

## Box II Observations where unity of invention is lacking (Continuation of item 2 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

see additional sheet

1. ☐ As all required additional search fees were timely paid by the applicant, this International Search Report covers all searchable claims.
  
2. ☒ As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.
  
3. ☐ As only some of the required additional search fees were timely paid by the applicant, this International Search Report covers only those claims for which fees were paid, specifically claims Nos.:
  
4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this International Search Report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

- ☐ The additional search fees were accompanied by the applicant's protest.
- ☐ No protest accompanied the payment of additional search fees.

FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

This International Searching Authority found multiple (groups of) inventions in this international application, as follows:

1. Claims: 1,2,5-9,11

Apparatuses and methods for the detection of an analyte in air comprising two enrichment stages and means for extraction of a material with a solvent.

2. Claims: 3,4,10

adsorption filter comprising a winding of resistance wire coated with a material having affinity for an analyte

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/EP 98/03531

Patent document cited in search report		Publication date	Patent family member		Publication date
US 5551278	A	03-09-1996	US	5092217 A	03-03-1992
			US	5099743 A	31-03-1992
			WO	9108466 A	13-06-1991
			US	5092219 A	03-03-1992
			US	5092155 A	03-03-1992
			US	5092218 A	03-03-1992
			US	5092157 A	03-03-1992
			US	5092156 A	03-03-1992
			US	5123274 A	23-06-1992
			US	5310681 A	10-05-1994
			US	5098451 A	24-03-1992
			EP	0501955 A	09-09-1992
<hr/>					
EP 0401861	A	12-12-1990	US	4987767 A	29-01-1991
			AT	153131 T	15-05-1997
			AU	633582 B	04-02-1993
			AU	5690590 A	13-12-1990
			CA	1329494 A	17-05-1994
			CA	2018697 A	09-12-1990
			DE	69030686 D	19-06-1997
			DE	69030686 T	11-09-1997
			DK	401861 T	03-11-1997
			ES	2102355 T	01-08-1997
			GR	3024358 T	28-11-1997
			IL	94684 A	29-12-1994
			IL	107672 A	15-03-1995
			JP	2119373 C	06-12-1996
			JP	3087629 A	12-04-1991
			JP	8033338 B	29-03-1996
			JP	2610118 B	14-05-1997
			JP	7209151 A	11-08-1995
			KR	9409049 B	29-09-1994
			US	5465607 A	14-11-1995
			US	5585575 A	17-12-1996
			US	5345809 A	13-09-1994
			AU	679387 B	26-06-1997
			AU	3063495 A	09-11-1995
			AU	7032291 A	18-07-1991
			CA	2070648 A	09-06-1991
			EP	0502998 A	16-09-1992

# INTERNATIONAL SEARCH REPORT

Information on patent family members

Int. l. Application No

PCT/EP 98/03531

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP 0401861 A		JP 9126964 A	16-05-1997
		JP 9126965 A	16-05-1997
		JP 9126966 A	16-05-1997
		WO 9109307 A	27-06-1991
		US 5109691 A	05-05-1992
US 4194884 A	25-03-1980	DE 2944774 A	12-06-1980
		FR 2442073 A	20-06-1980
		GB 2035125 A,B	18-06-1980
		JP 55074440 A	05-06-1980
		NL 7907940 A	28-05-1980
		SE 7909685 A	25-05-1980
US 4819477 A	11-04-1989	CA 1279207 A	22-01-1991
		EP 0236047 A	09-09-1987
		JP 6100521 B	12-12-1994
		JP 63079033 A	09-04-1988
US 5300758 A	05-04-1994	US 5108705 A	28-04-1992
		AT 134445 T	15-03-1996
		CA 2037862 A	13-09-1991
		DE 69117201 D	28-03-1996
		DE 69117201 T	19-09-1996
		EP 0447174 A	18-09-1991
		JP 4220558 A	11-08-1992
US 4128008 A	05-12-1978	NONE	
US 5138889 A	18-08-1992	NONE	
US 5395589 A	07-03-1995	CA 2120682 A	07-10-1995
		GB 2288234 A,B	11-10-1995
FR 2660873 A	18-10-1991	GB 2243447 A,B	30-10-1991
US 4909090 A	20-03-1990	CA 2015157 A	24-10-1990
		EP 0395194 A	31-10-1990
		JP 2296128 A	06-12-1990

**THIS PAGE BLANK (USPTO)**